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WADC TECHNICAL REPORT 54-87

## PROTECTIVE CERAMIC COATINGS FOR TITANIUM

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WRIGHT AIR DEVELOPMENT CENTER

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## PROTECTIVE CERAMIC COATINGS FOR TITANIUM

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*Power Plant Laboratory  
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## FOREWORD

This report, WADC Technical Report 54-87, was prepared by Herbert R. Toler, W. J. Plankenhorn, and Dwight G. Bennett at the University of Illinois in the Department of Ceramic Engineering as Report No. 67 under USAF Contract 33(616)-320, RDO No. 506-67, Ceramic Coatings for Aircraft Power Plants. It summarizes the results of a series of investigations with regard to the ability of ceramic coatings to protect titanium from oxidation and embrittlement at elevated temperatures. The technical phases of the contract are administered by the Power Plant Laboratory of the Wright Air Development Center with Lt. J. B. Hanover acting as Project Engineer.

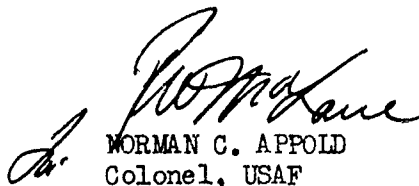
#### ABSTRACT

This final report summarizes the results of a series of tests conducted during an investigation of the effect of ceramic coatings on the physical and metallurgical changes occurring in titanium and/or its alloys when subjected to extended heating at temperatures of from 1400 to 1800°F. It was found that various coatings, originally designed for application to iron or low alloy content metals and stainless steels could be successfully applied to titanium. Such coatings furnished protection against oxidation at temperatures up to and including 1700°F as determined by weight increase determinations for uncoated and coated specimens. Properly selected coatings were shown to retard embrittlement of titanium at temperatures up to at least 1500°F as measured by impact resistance and indicated by metallurgical examination. Ceramic coatings prepared from frits which had been vacuum melted after the initial smelting and quenching were found to be the most effective in protecting the metal against embrittlement.

#### PUBLICATION REVIEW

The publication of this report does not constitute approval by the Air Force of the findings or the conclusions contained therein. It is published only for the exchange and stimulation of ideas.

FOR THE COMMANDER:

  
NORMAN C. APPOLD  
Colonel, USAF  
Chief, Power Plant Laboratory

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# PROTECTIVE CERAMIC COATINGS FOR TITANIUM

## I. INTRODUCTION

In this final report the work previously carried out (Ref. 1, 2, 3) under U. S. Air Force Contract 33(616)-320 is summarized and the results of all subsequent work presented.

### 1. Historical

Titanium and its alloys present very attractive commercial possibilities as engineering materials due to their ductility, high strength to weight ratio, and excellent corrosion resistance. However, titanium has been found to have certain definite disadvantages. These include the loss of strength and the embrittlement of the metal upon continued exposure to the atmosphere at temperatures above approximately 1000°F.

### 2. Scope

The present investigation was undertaken to explore the possibilities of retarding the embrittling high temperature reactions of titanium and/or its alloys with elements present in the atmosphere by the use of adherent ceramic coatings fired on to the metal surface. This work necessarily included a study of both coated and uncoated specimens and methods of evaluating their relative embrittlement.

### 3. Review of the Literature

While a vast amount of research has been reported on titanium and its alloys, the literature discloses very little information on protective coatings for titanium. Kluz, Kalinowski and Wehrmann (Ref. 4) have reported studies on the application of silicon, and silicide coatings containing either 57%Ni-43%Si or 50%Al-50%Si to titanium by sintering techniques at temperatures of 1000°C in a dried helium atmosphere. These investigators reported good performance in oxidation tests from specimens coated first with a layer of silicon metal and then with a layer of 50%Al-50%Si.

Suder (Ref. 5) reported attempts to produce an adherent glassy coating on titanium metal at 1200°F and 1500°F using titanium bearing enamels. He stated that little bond was produced between the metal and the coating at either temperature.

Work reported by Craighead, Lenning and Jaffee (Ref. 6) was found to be of considerable interest in the present investigation. They state that the line markings often associated with titanium and its alloys are due to an insoluble hydride of titanium. The authors indicate that the chief mechanical property

affected by this hydride phase is a significant lowering of the impact strength. The hydrogen content found in commercial alloys was reported to be in the range of 0.3 to 0.5 atomic per cent and was said to be sufficient to cause a substantial lowering of the impact energy absorption level without affecting the tensile properties.

Further work reported by Holden, Ogden and Jaffee (Ref. 7) indicates that the degree of dispersion of the titanium hydride has an important effect on impact energy values. When the hydride is finely dispersed as a result of quenching, the impact energy values are higher than when the hydride is present as line markings resulting from a slower cooling rate through the hydride precipitation range (200-300°C).

## II. EXPERIMENTAL DETAILS

### 1. Test Equipment

#### a. Controlled atmosphere furnace

A special furnace (Ref. 1) was constructed for the firing of titanium in argon and other gases. The firing chamber was formed by an "alundum" cylinder, of two inches inside diameter and six inches long. The system was so constructed as to enable it to be evacuated to between 50 and 75 microns of mercury before the introduction of the desired atmospheres. The system was flushed once with the selected gas and then evacuated and refilled again before firing. The furnace is pictured in Figure 1.

#### b. Impact tester

In order to determine the relative embrittlement of titanium specimens, a modified version of the Porcelain Enamel Institute Impact Tester (Ref. 1) was used. The impact head was made in accordance with specification E-23-47T of the American Society for Testing Materials. The machine, as modified, was suitable for the Izod type cantilever beam test using type Y specimens. It was designed to impact notched titanium rods 2 in long by 3/16 in. in diameter, but it was also found to be suitable for the testing of 0.050 in thick, unnotched plate specimens. The maximum striking force of this machine was 1.65 ft lbs. A photograph of the impact tester appears as Figure 2.

#### c. Tensile tester

A Tinius Olsen type of tensile tester was used in making tensile strength determinations. This machine had a

no-load head speed of 0.014 inches per minute.

## 2. Test Procedures

### a. Oxidation tests

Specimens 1.5 in x 0.5 in x 0.050 in. cut from sheet stock and other specimens 2 in long x 0.187 in. in diameter cut from rod stock were coated with University of Illinois coatings UI250-2 and UI418-1. They were given three periods of heating, 24, 48, and 72 hours at each of four increasing temperatures, 1400, 1500, 1600 and 1700°F. The gain in weight per square centimeter of surface area was determined and compared with the weight increase for similarly heated bare specimens.

### b. Tensile tests

Tensile tests were conducted at room temperature using threaded samples with an overall length of 3.295 inches and a machined testing diameter of 0.140 inches. (See Figure 3). The specimens were machined from 0.187 in diameter rod. Special adapter grips were made for fitting the small tensile specimens into the tensile machine.

### c. Impact tests

Notched impact specimens 2 in x 0.187 in cut from rod stock were used in the initial investigations. The specimens were notched with a standard threading tool on a metal lathe. The groove was machined to leave a cross section of metal of  $0.112 \pm 0.003$  in. in diameter. Such specimens were heated in air, argon and a vacuum of 75 microns of mercury for intervals of ten minutes to 72 hours at temperatures of from 1550 to 1700°F. Groups of notched cylindrical specimens were coated and heated and their degree of embrittlement compared with that for uncoated specimens similarly heated. When specimens were notched before heating difficulty was encountered in removing the coating from the coated specimens while the notched sections of the uncoated specimens were severely oxidized. Attempts were made to machine the rods after heat treatment, but they were unsuccessful because of the extreme hardness of the heat treated metal. Therefore, it was found to be more practical to use un-notched specimens 1.5 in x 0.5 in x 0.050 in cut from sheet stock. Such specimens were used to compare the degree of embrittlement resulting from varied heat treating procedures on uncoated and coated specimens.

### d. Metallographic examination

Numerous techniques have been devised for the

metallographic examination of titanium. The method used in this investigation, which was found to give satisfactory results, was as follows: The specimen was set in a clear mounting plastic material and rough polished on a rotating wheel using 240, 400 and 600 grades of silicon carbide paper. Water was originally used as a lubricant but a mixture of paraffin and oil was later found to be excellent for this purpose. The final polish was developed using water suspended alpha alumina of less than 0.5 micron size on a synthetic rayon type of cloth (Microcloth). The samples were then etched for 10 to 15 seconds and the polish repeated. Etching and polishing was usually repeated 3 times to bring out a satisfactory surface. The etchant used was that reported as "A etch" by Finlay, Resketo and Vordahl (Ref. 8) which consisted of:

- 1 part by volume of hydrofluoric acid
- 1 part by volume of concentrated nitric acid
- 2 parts by volume of glycerol

According to the authors, the hydrofluoric acid attacks the metal, the nitric acid brightens the surface by removing stain and residue, and the glycerol acts as a vehicle and moderator.

### 3. Metals Tested

#### a. Commercially pure titanium

Rod stock, 0.187 inches in diameter, from Allegheny Ludlum Heat No. 1949 was used to make up notched impact specimens and specimens for tensile testing. Specimens cut from sheet stock from two different heat numbers, Allegheny Ludlum No. X493 and Titanium Metals Corporation of America No. M-124, received in three different shipments were tested to determine their relative resistance to embrittlement resulting from extended heating.

#### b. Titanium alloy

Specimens of Rem-Cru titanium alloy RC-130A sheet stock, with 8% manganese as the alloying metal, were tested for embrittlement at 1400, 1500 and 1600°F.

### 4. Metal Preparation for Coating

In order to accomplish the objectives of this study, it was necessary to successfully apply ceramic coatings to titanium. Exploratory investigations indicated that high temperature resistant ceramic coatings developed for iron and its alloys could be applied to commercially pure titan-

ium. Some adherence was obtained with each of the coatings tested, including UI32-21, UI346-1, UI418-1, UI424-1 and a commercial, titanium-bearing, white finish coat enamel. This was true when coatings were applied to the metal cleaned with alcohol, but sandblasting greatly improved the adherence of all coatings. Pickling, however, with either a 10% solution of sulphuric acid or a 20% solution of hydrochloric acid, was found to be a satisfactory method for preparing the metal for coating and to give more reproducible results.

## 5. Ceramic Coatings

### a. Coatings from conventionally smelted frits

Regularly smelted frits which were quenched in water and wet milled for application in the usual manner were used in preparing the coatings for the initial investigation. These included University of Illinois frits UI32, UI250, UI418, UI412, and UI424 (Ref. 1, 2). A number of additional frits were also smelted following conventional practices. Coatings were prepared from these frits by wet milling. Representative frit formulas are presented in Table I while Table II lists mill batch formulas for coatings prepared from these frits.

### b. Coatings prepared from vacuum melted frits

University of Illinois frit No. 32, previously smelted and water quenched following standard practices, was vacuum melted to remove water and any occluded gases. For vacuum melting, the frit was placed in an alundum crucible which was then placed in an electrically heated pot furnace so constructed that it was gas tight and could be evacuated. The furnace was pumped down to a vacuum of 75 microns of mercury while at room temperature and then rapidly heated to 1600°F. This temperature was maintained for one hour. The vacuum pump was operated throughout the heating period and until the furnace and frit charge had cooled to a temperature of 500°F or less. The cooled frit showed evidence of frothing resulting in the formation of a cellular structure with many large interconnected bubbles throughout the mass. The frit seemingly softened sufficiently to allow removal of the trapped gases without becoming fluid. The vacuum melted frit was dry ground to pass a 200 mesh sieve. Since mill batch water seems to be completely driven off during drying and firing (Ref. 9), the preground frit was milled with water as the vehicle and with calcined diaspore and bentonite as mill additions.

Two milled coating slips, UI32-51 and UI32-52 were prepared as per the batch formulas given in Table III. They were applied by dipping to 1.5 in x 0.5 in x 0.050 inch speci-

mens of titanium Ti-75A. The coated specimens were air dried and then fired for 10 minutes in air in an electrically heated furnace operated at 1675°F. The coated specimens were divided into two groups. One group was tested for impact resistance as coated and the other after extended heating.

#### c. Clear glass frits

Three new coating frits, UI496, UI497, and UI498 (See Table III) were formulated and smelted. These frits were based on the melted composition of UI32 frit except that in each case the glass coloring and so called adherence promoting oxides of cobalt, nickel and manganese were omitted. They were vacuum melted and milled into coating slips following accepted wet milling practices. These coatings were prepared to, 1) determine whether the oxides noted were important in this particular coating frit for titanium, and 2) to study the effects of increased refractoriness of the frit.

### III. RESULTS

#### 1. Oxidation Protection Provided by Ceramic Coatings

Ceramic coatings were found to offer protection to titanium against oxidation as determined by the relative increase in weight of coated and uncoated specimens. Uncoated titanium specimens heated for periods ranging from 24 to 167 hours at temperatures of 1400, 1500, 1600 and 1700°F showed definite weight increases due to oxidation. The rate of oxidation increased with increasing temperatures and the degree of oxidation with the length of time the specimens were heated at any given temperature. Ceramic coatings noticeably reduced the oxidation of the metal. University of Illinois coating UI418-1 was found to be more effective than UI250-2. As may be noted in Table I, Report No. 63, bare metal showed a weight gain of 3.40 milligrams per square centimeter after 72 hours heating at 1400°F. With coating UI250-2 the weight gain was 0.047 milligrams. With coating UI418-1, used in two tests of 72 hours at 1400°F, the weight gains were 0.00 and 0.018 milligrams per square centimeter, respectively.

As shown in that table, weight gains increased with increasing test temperatures but the relation among bare metal and specimens coated with UI250-2 and UI418-1 remained the same.

#### 2. Tensile Strength of Titanium

##### a. As received

Tensile tests conducted at room temperature gave an

ultimate strength of 86,200 psi for a commercially pure titanium Ti-75A (from Allegheny Ludlum Heat No. 1949). Heating the metal for as short a time as 10 minutes at a temperature of 1600°F apparently relieved the stresses in the metal as indicated by increased ductility and a reduction in the ultimate strength to 80,000 psi. (cf Table II Report 65). After 100 hours of heating at 1400°F the ultimate strength decreased further to about 78,000 psi with brittle fracture being very evident. Such fracture occurred with less reduction in cross-sectional area although per cent elongation did not change appreciably. The reasons for this unusual relationship between elongation and cross-sectional area are not known.

#### b. After ceramic coating

Ceramic coatings exerted some small but definite improvement in the ultimate tensile strength of commercially pure titanium after extended heating as shown herewith:

<u>Coating</u>	<u>Heating time</u>	<u>Temp., °F</u>	<u>Ult ts psi</u>
None	100 hrs	1400	78,000
UI493-1	" "	"	80,000
UI32-22	" "	"	80,400
UI424-1	" "	"	82,400
UI418-1	6 min(1)	1800	84,900

- (1) Specimens coated with UI418-1 were not heated for 100 hours at 1400°F since it was found that the high initial firing temperature produced severe embrittlement even though tensile strength was improved. These data are taken from Table II of Report No. 65.

### 3. Impact Strength of Titanium

#### a. Notched specimens

Notched specimens cut from 0.187 in diameter titanium Ti-75A bar stock were broken by impact after being variously heated. Heating for short periods of time at 1550°F (below the allotropic transformation) and at 1675°F (above the allotropic transformation) was found to increase the impact strength of bare metal specimens. This was presumably due to stress relief. However, prolonged heating at either temperature produced embrittlement. (cf Table II Report 63).

#### b. Variations between mill heats of titanium Ti-75A sheet stock

The metal from two different mill heats of titanium



Ti-75A sheet stock was used in the investigation of the effects of extended heating on the physical properties of the metal. A large variation in the impact resistance of the metal from the two different heats was noted. These differences are tabulated in Table IV. The metal from Allegheny Ludlum Heat No. X493 became very brittle after being heated for 100 hours at 1400°F. That from Titanium Metals Corporation of America Heat No. M-124, did not evidence embrittlement after being heated for 100 hours at 1450°F. The variation in the rate of embrittlement of titanium Ti-75A from the different heats may have been due in part to small differences in the composition which is reported (Ref. 10) to be nominally 0.10%Fe, 0.02%N, trace of O, less than 0.04C and 0.03W with the remainder being Ti.

Some differences were noted in the microstructure of the two heats of metal, as received. These differences can be seen in photomicrographs (a) and (b) of Figure 4. It may be noted that the grain size in the metal of heat No. X493 appears to be considerably finer than the grains in the metal from the heat No. M-124. The impurities in the first lot (No. X493) are also more evenly dispersed. The finer grain size of the material in that heat is even more noticeable in (c) and (d) of Figure 4 which show the structure of the metal after 100 hours of heating at 1400°F. The reason for the finer grain size is not known but it may be due to the inhibiting action of impurities.

#### c. Embrittlement as a function of time and temperature

Tests were conducted on specimens heated in air for various periods of time at temperatures of from 1400°F to 1850°F to determine the effects of time and temperature on the embrittling rate of titanium. The results, as tabulated in Table V, show that over 200 hours of heating at 1400°F were required to produce measurable embrittlement in titanium Ti-75A from heat No. M-124. This time dropped to 75 hours at 1500°F and to less than 1 hour at 1700°F. At 1800°F embrittlement occurred in less than 15 minutes. At 1850°F it occurred in less than 6 minutes. These data indicated that long heat tests in air at temperatures of less than 1500°F were impractical, with this particular stock, because of the extremely long periods of time required for testing and that the ceramic coating of this material in an air atmosphere at temperatures of 1700°F, or above, could not be recommended due to a considerable loss in ductility during the firing cycle.

#### d. Embrittlement of a titanium alloy

Titanium alloy RC-130A, with 8% manganese as the alloying metal, became extremely brittle after being heated for 30 minutes at 1400°F and after only 10 minutes at 1500°F. Firing in a partial vacuum of 75 microns reduced the degree of embrittlement resulting from 10 minutes of heating at 1500°F,

but did not eliminate it completely. The data from this series of tests are given in Table VI. Further work with titanium alloy RC-130A was not considered to be advisable at the present time because of the low temperature at which it embrittled.

#### 4. Effect of Ceramic Coatings on Impact Strength

##### a. Coatings using quenched frits

A series of 20 coatings, previously found to be suitable for the coating of iron and steels, were selected for a study of their ability to protect titanium from embrittlement. These coatings were prepared and applied to unnotched impact specimens cut out of sheet stock from heat No. X493. Uncoated and coated specimens were heated for 100 hours at 1400°F. The uncoated specimens showed considerable embrittlement. As noted in Table VII, and Figure 5, the specimens coated with University of Illinois coatings UI32-22 and UI493-1 showed the greatest improvement in impact resistance over the uncoated specimens. However, even with these coatings the results were scattered.

From these results it appeared that factors other than the melted constituents of the coating might be playing an important role in the protection of titanium.

##### b. Coatings using quenched and vacuum melted frits

The detrimental effects of hydrogen on the impact strength of titanium was previously noted (Ref. 6, 7). This together with the fact that water-free enamel frits have been shown to greatly reduce hydrogen produced defects in porcelain enamels applied to steel (Ref. 9) led to an investigation of vacuum melted frits for the production of ceramic coatings for application to titanium.

Coatings made up with UI32 vacuum melted frit (cf Sec. II, 5b) were found to greatly increase the resistance of titanium to impact. Bare metal specimens of titanium Ti-75A, heat No. M-124, showed considerable embrittlement after being heated for 75 hours at 1500°F. Similar specimens when coated with UI32-52 were found to retain their ductility after 250 hours of heating at the same temperature. (250 hours was the longest period of heating used in this series of tests). These data are recorded in Table VIII. Photographs of representative specimens are shown in Figure 6. When similar specimens were sectioned, marked differences were noted between the microstructures of the coated samples which remained ductile, and the embrittled bare specimens. These differences can be noted in photomicrographs (a) and (b) of Figure 7, taken at 200X. Photomicrographs (a) and (b) of Figure 8, taken at 75X show the

considerable difference in cross-section between the coated and uncoated samples due to severe oxidation of the uncoated ones.

Coatings prepared from vacuum melted No. UI32 frit proved to be more consistently effective in protecting commercially pure titanium from embrittlement than coatings milled with any of the conventionally processed frits. Coatings prepared from conventionally processed frits, and applied to titanium, undoubtedly serve as a barrier to the embrittling elements of the atmosphere but they themselves contain moisture and other harmful gases which can react with the metal during the maturing fire. Vacuum melting of the frit appears to remove most of these gases.

#### c. Coatings using vacuum melted clear glass frits

The coatings UI496-2, UI497-2, and UI498-2 prepared from the three frits which were cobalt, nickel and manganese free produced a fair bond between the coating and the metal upon initial fire. This bond deteriorated rapidly, however, when the coated specimens were heated for prolonged periods of time at 1500°F. The data in Table VIII show that less protection was obtained with these coatings than from UI32-51 and UI32-52. A direct comparison can be made between specimens coated with UI32-52 and UI496-2, which were identical except for the adherence oxides.

### IV. CONCLUSIONS

Various coatings, originally designed for application to iron or low alloy content metals and stainless steels of the 18-8 type can be successfully applied to titanium.

Ceramic coatings effectively retard the oxidation of titanium subjected to elevated temperatures.

Properly selected ceramic coatings definitely retard embrittlement of titanium at temperatures up to at least 1500°F. Coatings prepared from frits which have been smelted and quenched and then reheated in a vacuum are most effective.

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TABLE I. - REPRESENTATIVE FRIT FORMULAS

## 1. Raw batch weights

Frit No.	UI32	UI250	UI346	UI412	UI493	UI496	UI497	UI498
Raw Material								
Potash Feldspar	34.2	15.0	47.4	72.7	35.8	40.40	---	---
Quartz	24.8	29.0	18.3	---	24.9	28.10	53.90	48.20
Borax	23.8	25.0	17.9	---	24.5	---	---	---
Boric Acid	6.4	---	---	---	---	---	---	---
Soda Ash	4.1	---	6.1	9.1	6.6	6.90	8.00	8.00
Fluorspar	3.7	---	2.8	---	3.9	4.34	4.28	4.28
Cobalt Ox.	0.5	---	0.37	---	---	---	---	---
Nickel Ox.	0.5	---	0.37	---	---	---	---	---
Manganese Diox.	1.5	---	1.12	---	---	---	---	---
Soda Nitre	---	3.0	4.4	9.1	4.3	5.56	6.40	6.40
Titanium Diox.	---	12.0	---	---	---	---	---	---
Calcium Carb.	---	1.5	---	---	---	---	---	---
Sodium Antimonate	---	0.2	---	---	---	---	---	---
Aluminum Hydrate	---	1.0	---	---	---	---	12.88	18.58
Zinc Ox.	---	0.8	---	---	---	---	---	---
Cryolite	---	6.5	---	---	---	---	---	---
Magnesium Carb.	---	6.0	---	---	---	---	---	---
Vanadium Pentox.	---	---	1.25	9.1	---	---	---	---
Anhydrous Borax	---	---	---	---	---	14.70	14.54	14.54
Total	99.5	100.0	100.01	100.0	100.0	100.00	100.00	100.00

## 2. Oxide compositions, per cent

## Oxide

SiO <sub>2</sub>	56.5	47.2	57.1	52.7	58.2	58.20	58.20	52.40
Al <sub>2</sub> O <sub>3</sub>	8.4	4.5	10.8	16.4	8.6	8.62	13.90	20.00
B <sub>2</sub> O <sub>3</sub>	10.5	11.2	8.0	---	10.9	10.85	10.85	10.85
Na <sub>2</sub> O	12.1	6.8	10.9	11.7	12.4	12.40	12.40	12.40
K <sub>2</sub> O	5.1	2.2	6.7	10.0	5.3	5.30	---	---
CaF <sub>2</sub>	4.5	---	3.4	---	4.6	4.63	4.65	4.65
CoO	0.6	---	0.45	---	---	---	---	---
NiO	0.6	---	0.45	---	---	---	---	---
MnO <sub>2</sub>	1.8	---	1.35	---	---	---	---	---
CaO	---	1.0	---	---	---	---	---	---
ZnO	---	1.0	---	---	---	---	---	---
MgO	---	3.4	---	---	---	---	---	---
TiO <sub>2</sub>	---	14.6	---	---	---	---	---	---
Sb <sub>2</sub> O <sub>5</sub>	---	0.2	---	---	---	---	---	---
Na <sub>3</sub> AlF <sub>6</sub>	---	7.9	---	---	---	---	---	---
V <sub>2</sub> O <sub>5</sub>	---	---	1.2	9.2	---	---	---	---

Total	100.1	100.0	100.35	100.0	100.0	100.00	100.00	100.30
-------	-------	-------	--------	-------	-------	--------	--------	--------

Calculated Co-  
efficient of  
Expansion  
(Cubical) X10<sup>7</sup>

272	282	287	336	271	271	253	279
-----	-----	-----	-----	-----	-----	-----	-----

TABLE II. - SUMMARY OF MILL BATCH FORMULAS

Coating No.	UI32-21	UI32-22	UI346-1	UI412-2	UI424-1	UI493-1
Material:						
Frit	100	88	100	55	100	100
Enamellers Clay	7	7	7	7	7	6
Borax	0.75	0.75	0.75	0.75	---	---
Diaspore						
(1st Grade)	---	12	---	---	---	---
Potash Feldspar	---	---	---	45	---	---
Water	50	50	50	50	45	50

TABLE III. - MILL BATCH FORMULAS FOR CERAMIC COATINGS USING VACUUM  
MELTED FRITS

Coating No.	UI32-51	UI32-52	UI496-2	UI497-2	UI498-2
Parts by weight					
Frit No. UI32	88	80	--	--	--
Frit No. UI496	--	--	100	--	--
Frit No. UI497	--	--	--	100	--
Frit No. UI498	--	--	--	--	100
Calcined Diaspore (1)	12	20	20	10	5
Bentonite	2	2	2	1.75	1.5
Water	55	55	45	47.5	52

(1) Preground to pass 100 mesh.

TABLE IV. - EFFECT OF 100 HOURS HEATING ON THE IMPACT RESISTANCE OF  
TWO MILL HEATS OF TITANIUM T1-75A

Ship No.	Heat No.	Heating temp °F	Impact energy <sup>(1)</sup> absorbed, ft lbs	Comments
One	X-493	1400	0.87	Brittle
Two	M-124	1400	1.65	Ductile
"	"	1450	1.65	Ductile
"	"	1500	0.30	Brittle, heavy scale
Three	M-124	1400	1.65	Ductile
"	"	1450	1.61	Start of embrittlement
"	"	1500	0.21	Brittle, heavy scale

(1) Average of four tests. Impact tests were conducted on unnotched specimens of sheet stock 1.5 in x 0.5 in x 0.050 in. The maximum energy output of the machine was 1.65 ft lbs. When this value is shown it indicates that the specimens were bent but not broken.



TABLE V. - THE EMBRITTLEMENT OF TITANIUM Ti-75A AS A FUNCTION OF  
TIME AND TEMPERATURE

time	Heating <sup>(1)</sup> temp °F	Impact energy <sup>(2)</sup> absorbed, ft lbs	Comments
200 hr	1400	>1.65	Ductile
50 "	1500	>1.65	Ductile
75 "	"	1.63	Start of embrittlement
100 "	"	0.30	Brittle
.5 hr	1700	>1.65	Ductile
1 "	"	0.85	Brittle
10 min	1800	>1.65	Ductile
15 "	"	1.17	Brittle
3 "	1850	>1.65	Ductile
6 "	"	0.56	Brittle

(1) Heated in air.

(2) Average of four tests. Impact tests were conducted on unnotched specimens of sheet stock 1.5 in x 0.5 in x 0.050 in. The maximum energy output of the machine was 1.65 ft lbs. When this value is shown it indicates that the specimens were bent but not broken.

TABLE VI. - EFFECT OF TEMPERATURE ON THE EMBRITTLING RATE OF  
TITANIUM RC-130A

time	Heating temp °F	atmos	Impact energy <sup>(1)</sup> absorbed, ft lbs	Comments
10 min	1400	air	>1.65	Ductile
30 "	"	"	1.14	Brittle
10 "	1500	"	0.12	Brittle, heavy scale
10 "	"	vac <sup>(2)</sup>	1.28	Brittle
10 "	1600	air	0.12	Brittle, heavy scale

- (1) Average of four tests. Impact tests were conducted on unnotched specimens of sheet stock 1.5 in x 0.5 in x 0.050 in. The maximum energy output of the machine was 1.65 ft lbs. When this value is shown it indicates that the specimens were bent but not broken.
- (2) Specimens heated for 10 minutes at 1500°F in a partial vacuum of 75 microns of mercury.

TABLE VII. - THE EFFECT OF CERAMIC COATINGS ON THE IMPACT STRENGTH  
OF UNNOTCHED SPECIMENS OF TITANIUM Ti-75A HEATED FOR 100 HOURS AT  
1400°F

Coating No.	Firing time min	temp °F	Impact energy <sup>(1)</sup> absorbed, ft lbs	Comments
Uncoated	--	--	0.87	Brittle fracture
UI32-39	6	1600	.64	" "
UI318-1	3	1600	.64	" "
UI490-1	6	1650	.70	" "
UI32-21	6	1600	.72	" "
UI436-1	6	1600	.73	" "
UI339-1	3	1600	.75	" "
UI485-1	6	1650	.75	" "
UI437-1	8	1600	.78	" "
UI418-7	6	1650	.82	" "
UI497-1	6	1650	.82	" "
UI460-1	6	1650	.92	" "
UI90-10	6	1600	.96	" "
UI181-1	6	1600	.96	" "
UI474-1	6	1650	.96	" "
UI483-1	6	1650	.98	" "
UI495-1	10	1650	1.06	" "
UI494-1	10	1650	1.10	" "
UI468-1	6	1650	1.28	" "
UI32-22	10	1650	>1.49 <sup>(2)</sup>	7 of 14 did not break
UI493-1	10	1650	>1.51 <sup>(2)</sup>	2 of 4 did not break

(1) Average of four or more tests. Impact tests were conducted on specimens 1.5 in x 0.5 in x 0.050 in cut out of sheet stock from heat No. X493. The maximum energy output of the machine was 1.65 ft lbs.

(2) Nominal averages including specimens which did not break up to the capacity of the machine. Actual averages would be somewhat higher.

TABLE VIII. - THE EFFECT OF CERAMIC COATINGS, PREPARED FROM VACUUM  
MELTED FRITS, ON THE IMPACT STRENGTH OF UNNOTCHED SPECIMENS OF  
TITANIUM T1-75A AFTER EXTENDED HEATING AT 1500°F IN AIR

Specimen condition	Coating No.	Frit type	Hours heated	Energy <sup>(1)</sup> absorbed	Comments
Uncoated	---	---	0	>1.65	Ductile
"	---	---	50	>1.65	"
"	---	---	75	1.63	Start of embrittlement
"	---	---	100	0.30	Brittle
"	---	---	250	0.12	Brittle, heavy scale
Coated	UI496-2	Clear	100	1.11	Brittle, poor adherence
"	UI497-2	Clear	100	1.09	" " "
"	UI498-2	Clear	100	1.09	" " "
"	UI32-51	Colored	100	>1.65	Ductile, fair adherence
"	UI32-52	Colored	100	>1.65	Ductile, good adherence
"	UI32-52	Colored	250	>1.65	Ductile, " "

(1) Impact energy absorbed, ft lbs. Average of four tests. Impact tests were conducted on unnotched specimens 1.5 in x 0.5 in x 0.050 in cut out of sheet stock from heat No. M-124. The maximum energy output of the machine was 1.65 ft lbs. When this value is shown it indicates that the specimens were bent but not broken.

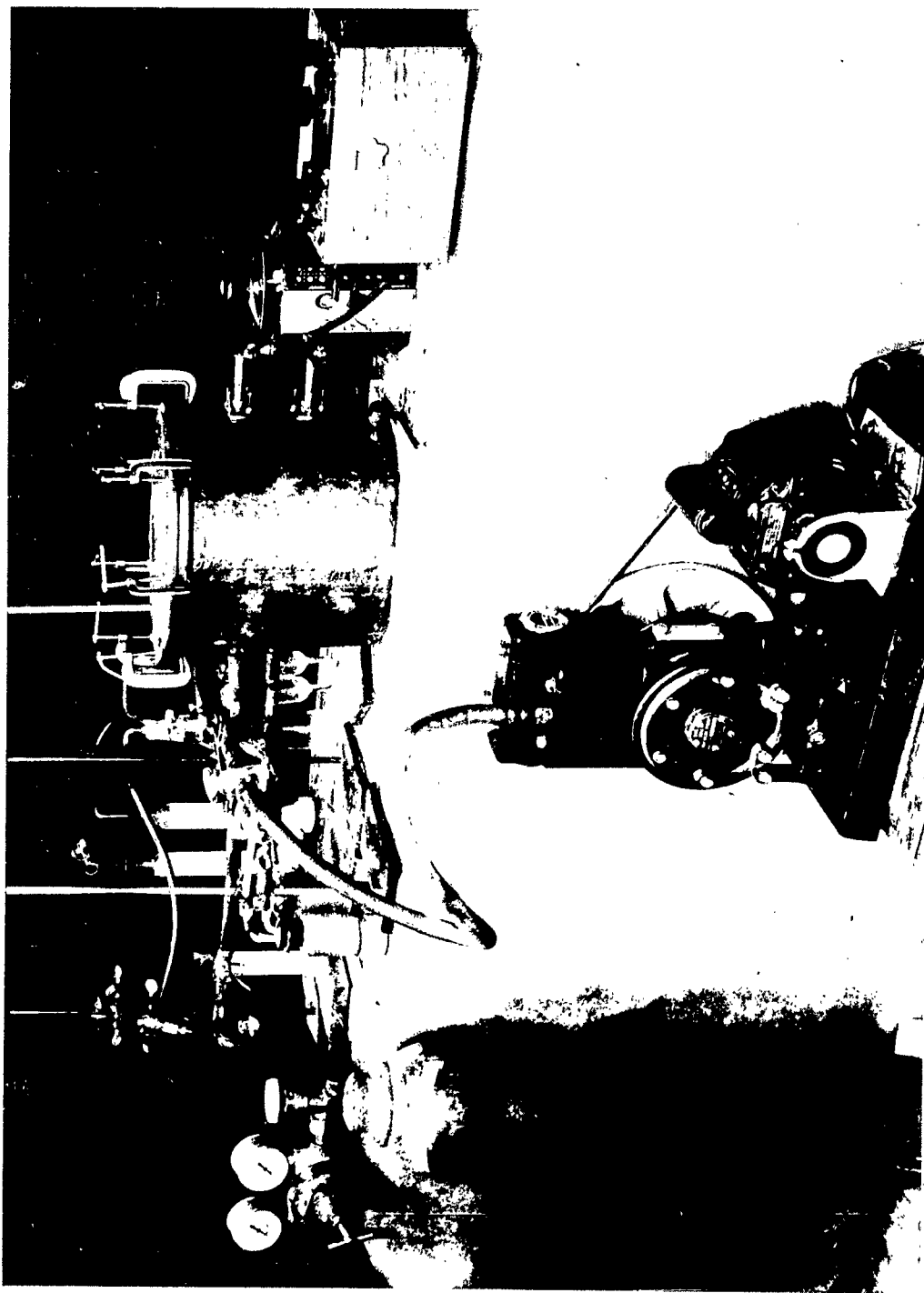


Figure 1. - Controlled atmosphere furnace used in developing protective ceramic coatings for titanium.

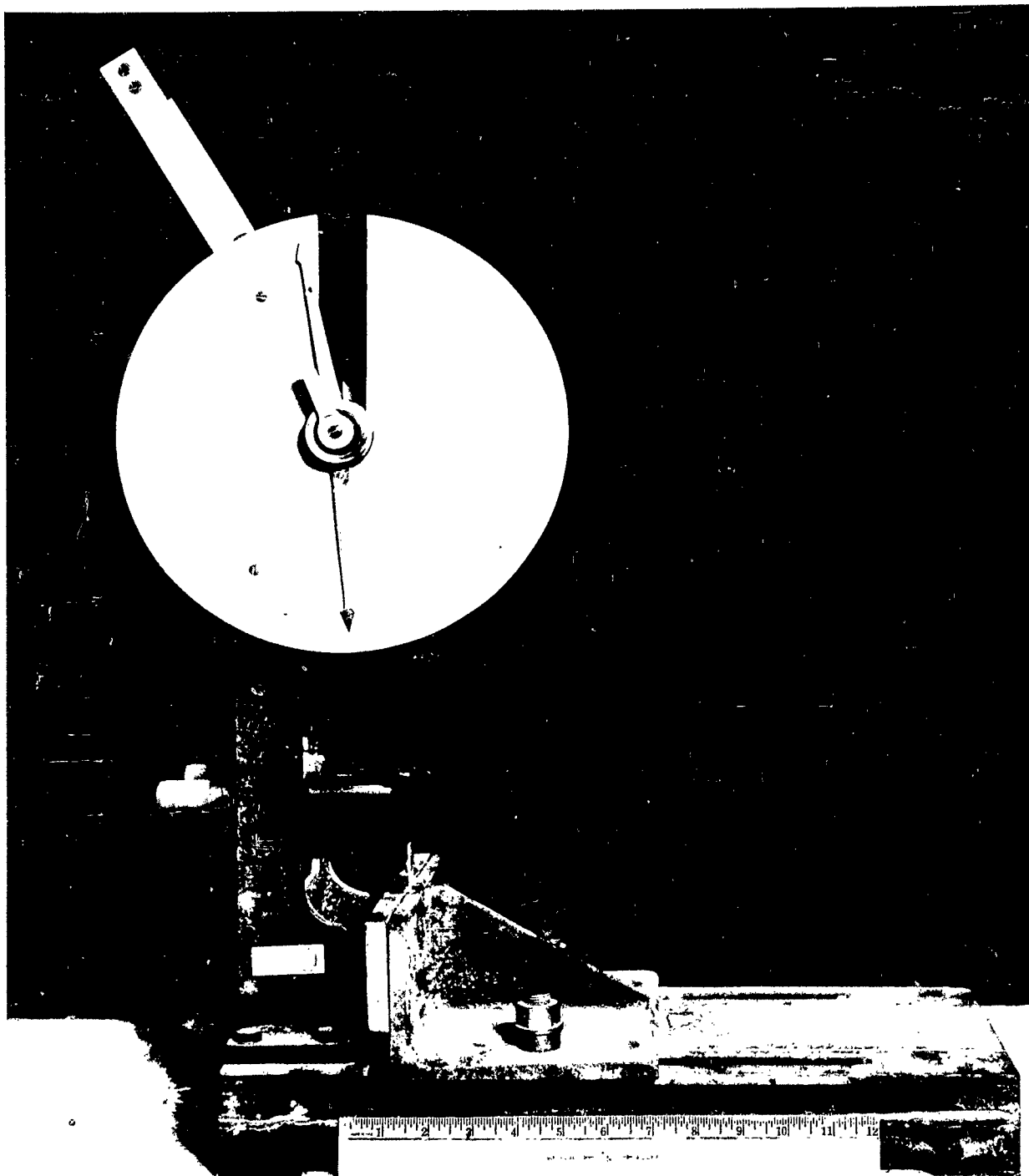
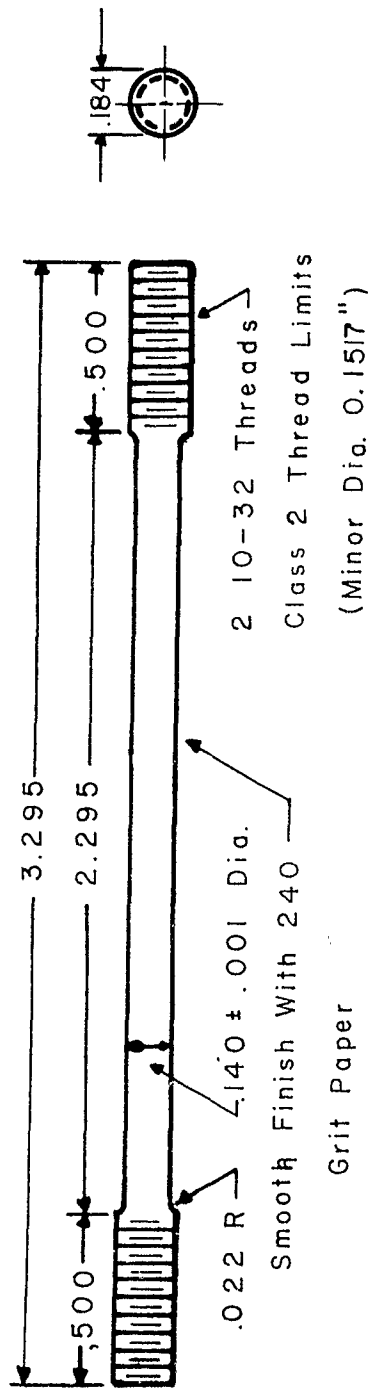


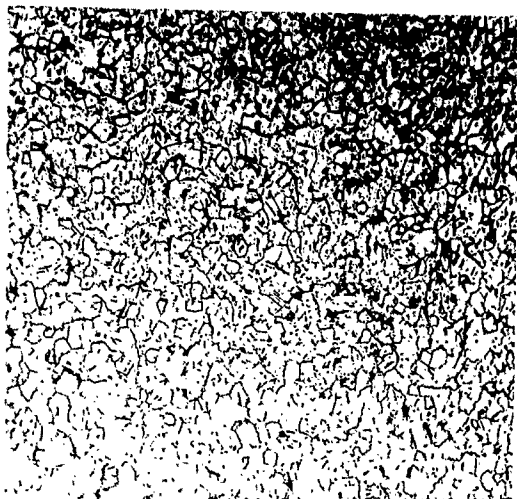
Figure 2. - Impact tester, showing the striking head and a specimen of sheet stock in the test position.



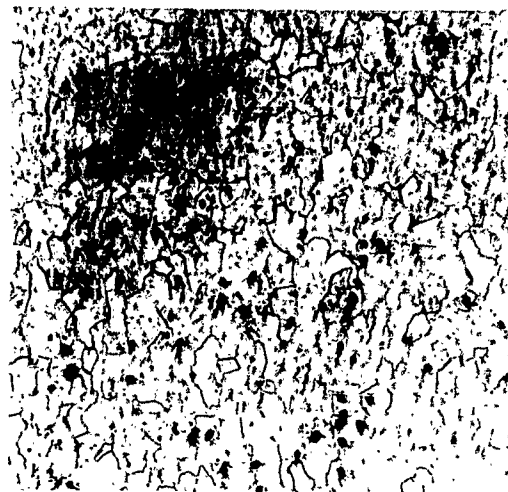
Material: Titanium

Scale: 2" = 1"

Figure 3. - A tensile test specimen designed for use in a Tinius Olsen machine.



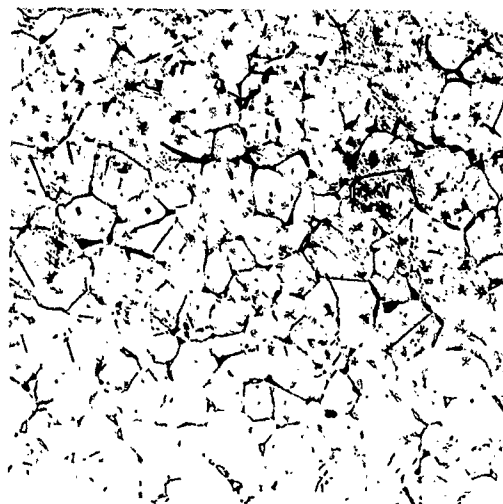
(a) Allegheny Ludlum Heat  
No. X493 as received



(b) Titanium Metals Corp.  
of America Heat No.  
M-124 as received



(c) Allegheny Ludlum Heat  
No. X493 after heating



(d) Titanium Metals Corp.  
of America Heat No.  
M-124 after heating

Figure 4. - Photomicrographs of sections from two different heats of titanium Ti-75A, as received and after heating for 100 hours at 1400°F. 200X Type "A" etchant.



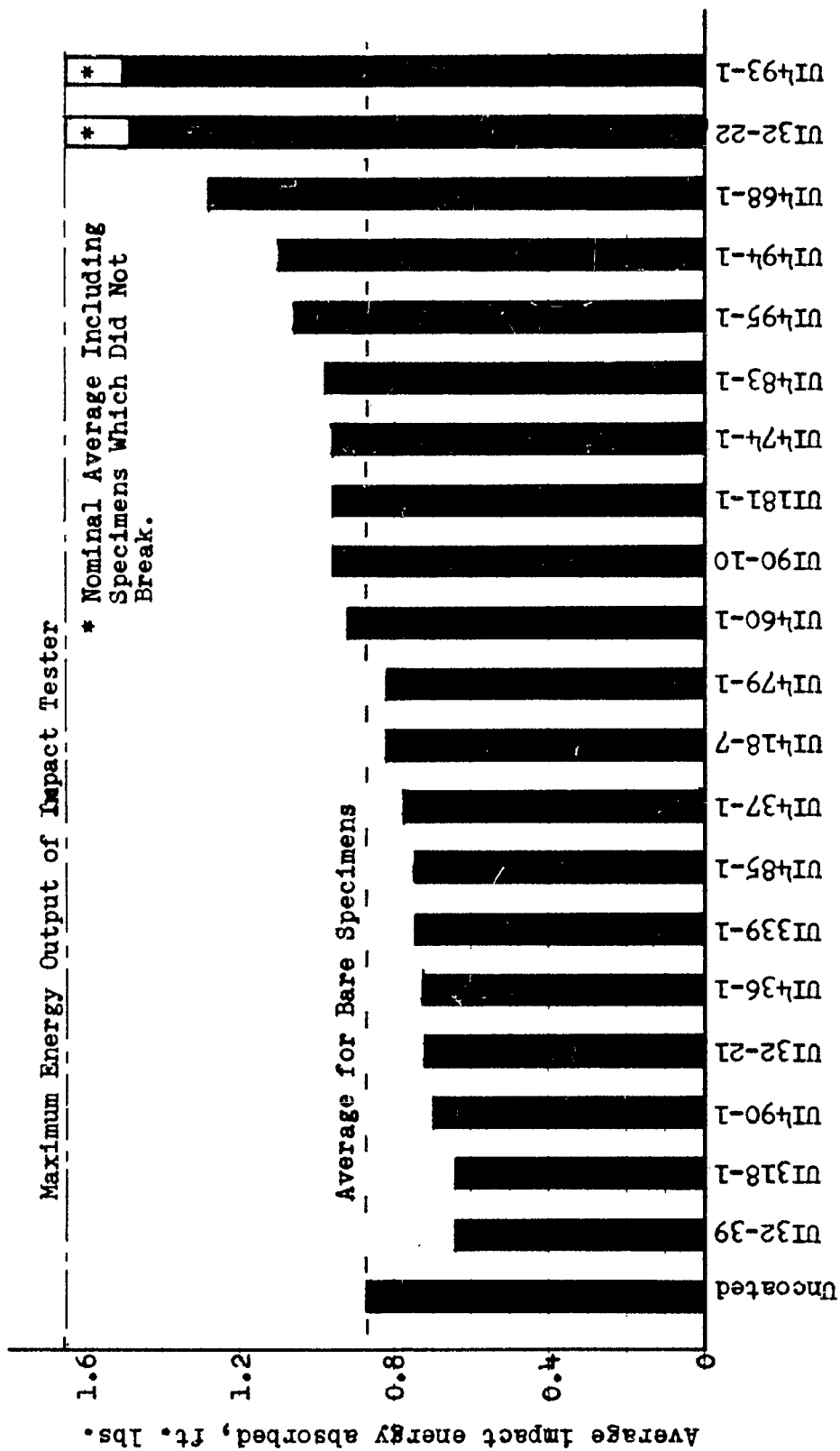


Figure 5. - The effect of various ceramic coatings on the embrittlement of titanium Ti-75A which had been subjected to 100 hours of heating at 1400°F.

University of Illinois coating No.

Uncoated specimens



Heating time, hours

None

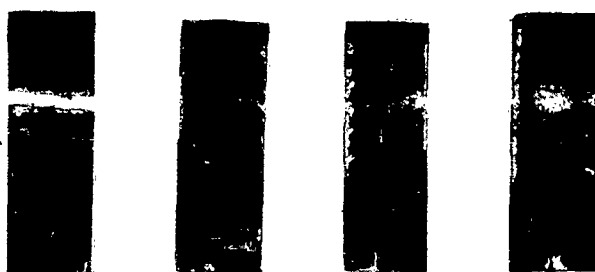
50

75

100

250

Coated specimens



Ceramic coating  
Heating time, hours

UI32-22  
100

UI32-51<sup>(1)</sup>  
100

UI32-52<sup>(1)</sup>  
100

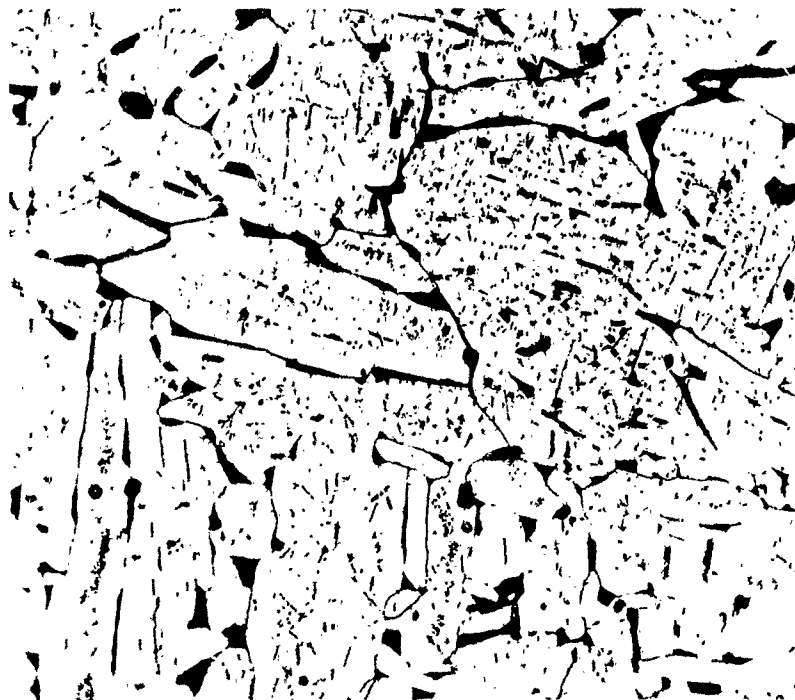
UI32-52<sup>(1)</sup>  
250

(1) Ceramic coatings prepared with vacuum melted frit.

Figure 6. - Specimens of uncoated and ceramic coated titanium impact tested after heating for various times at 1500°F.



(a) brittle



(b) ductile

**Figure 7.** - Photomicrographs of (a) uncoated and (b) ceramic coated (UI32-52) specimens of titanium Ti-75A after heating for 250 hours at 1500°F. 200X Type "A" etchant.



(a) brittle. Section reduced by oxidation



(b) ductile. Section maintained by coating

Figure 8. - Photomicrographs of (a) uncoated and (b) ceramic coated (UI32-52) specimens of titanium Ti-75A after heating for 250 hours at 1500°F. 75X Type "A" etchant.

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- 1 GE Co, Thomson Lab, 920 Western Ave, West Lynn 3, Mass, Attn: W. Badger
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- 1 Gladding McBean & Co, 2901 Los Feliz Blvd, Los Angeles 39, Cal, Attn: W. O. Brandt
- 1 Glascote Prod, Inc, 20900 St. Clair Ave, Cleveland 17, O, Attn: Mr. L. Breeze
- 2 Henry L. Crowley & Co, Inc, 1 Central Ave, W. Orange, N. J., Attn: H. I. Danzieger
- 1 Kearfott Co, Inc, 1150 McBride Ave, Little Falls, N. J.
- 2 Lorain Metal Treating Co, 1915 E. 28th St, Lorain, O, Attn: Mr. W. F. Stark
- 1 Metallurgical R & D Co, Inc, 910 - 17th St, N. W., Washington 6, D, C, Attn: Mr. Taylor
- 1 Michigan Chemical Corp, St. Louis, Mich, Attn: Jack Allen Campbell
- 1 Nat'l Carbon Research Labs, P. O. Box 6087, Cleveland 1, O, Attn: Dr. H. MacPherson
- 1 Nat'l Day Co, 300 W. 69th St, Chicago 21, Ill, Attn: J. P. Novak
- 1 Nat'l Lead Co, Niagara Falls, N. Y.
- 1 Norton Co, Worcester 6, Mass, Attn: L. H. Milligan
- 1 O. Hommel Co, Box 475, Pittsburgh 30, Pa, Attn: W. T. Campbell
- 1 Pemco Corp, 5601 Eastern Ave, Baltimore 24, Md.
- 1 Pfaunder Co, 1000 W. Ave, Rochester 3, N. Y., Attn: Mr. S. C. McCann

Industry (Cont'd)

- 1 P. R. Mallory & Co, Inc, 3029 E. Washington St, Indianapolis 6, Ind, Attn: Mr. Swazy
- 1 Raytheon Mfg Co, Government Contracts Dept, 100 River St, Waltham, Mass, Attn:  
Mr. Hurff
- 1 Seaporcel Metals, Inc, 28-20 Borden Ave, L. I. City, N. Y., Attn: Mr. P. S. Cecil
- 1 Ordnance Dept, Springfield Armory, Springfield, Mass.
- 1 Stewart-Warner Corp, 1514 Drover St, Indianapolis 7, Ind, Attn: Mr. A. Gelb
- 2 Stupakoff Ceramic & Mfg Co, Latrobe, Pa, Attn: R. E. Stark
- 1 Thompson Prod, Inc, 23555 Euclid Ave, Cleveland 17, O, Attn: A. T. Colwell
- 1 Union Carbide and Carbon Corp, Haynes Stellite Co, 725 S. Lindsay St, Kokomo, Ind,  
Attn: W. O. Sweeny
- 1 Universal-Rundle Corp, Box 960, New Castle, Pa, Attn: Dr. A. L. Johnson
- 2 Wall Colmonoy Corp, 19345 John R St, Detroit 3, Mich, Attn: Messrs. LaRou and Peaslee
- 4 Westinghouse Electric Corp, Third National Bank Bldg, Dayton, O, Attn: Mr. Hodges
- 1 Hotpoint Co, 4769 W. Electric Co, Milwaukee 14, Wis, Attn: C. J. Carter